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ABSTRACT

This document is an instructional module package prepared in objective form for use by an instructor familiar with the spectrophotometric analysis of total iron, filtrable iron and ferrous iron in a water supply. Included are objectives, an instructor guide, student handouts, and transparency masters. A video tape is also available from the author. This module considers use of the spectrophotometer, Beer's Law, calibration curves, preparation of reagents and standards, test procedures and interferences.
(Author/RH)

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DETERMINATION OF IRON IN WATER

Training Module 5.210.2.77

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September, 1977

SE 024 154

Module No:	Module Title:
	The Determination of Iron in Water.
Approx. Time:	Submodule Title:
	Topic:
5 hours	Summary

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Given the proper equipment and reagents, determine the concentration of total iron, filtrable iron, and ferrous iron spectrophotometrically.
2. Describe interferences and possible sources of error in an iron analysis.
3. Describe safety precautions which should be employed in performing the analysis.
4. Explain why the determination should be performed and how the test results will be used.

Instructional Aids:

1. Fe/Mn Video tape
2. Overhead Transparencies (FeI-Fe9)

Instructional Approach:

Lecture, Demonstration, Discussion, Video tape viewing, practical.

References:

1. W.H.Nebergall, F.C.Schmidt, H.F.Holtzclaw, Jr. "College Chemistry with Qualitative Analysis." 5th ed., Lexington, Mass. D.C.Heath, 1976.
2. "Standard Methods for the Examination of Water and Wastewater", 14th Edition.
3. Operator's Manual for the Bausch and Lomb Spectronic 20 Spectrophotometer.

Class Assignments:

Module No:	Module Title: The Determination of Iron in Water
Approx. Time: 0.5 hours	Submodule Title: Topic: Introduction to the Chemistry of Iron

Instructional Objective:

Upon completion of this module, the participant should be able to:

1. Locate iron on the periodic chart of the elements.
2. Classify iron as an element.
3. Identify the three major oxidation states of iron.
4. Name and list the formula of four different compounds of iron.

Instructional Aids:

Transparency Fe-periodic chart of the elements.

Instructional Approach:

Lecture/discussion

References:

Nebergall, et al, pp. 898-910.

Class Assignments:

None

Module No: Fe	Topic: Introduction of Chemistry of Iron
Instructor Notes:	Instructor Outline:
Transparency Fe-1 Periodic Chart	1. Nature is made up of elements. Each element has an atomic symbol to represent the element and its atoms. All elements are listed on periodic chart of the elements. Iron is one such element Its atomic symbol is Fe. 2. Iron is a metal. Iron is a transition metal. Some compounds of transition metals are colored. 3. Iron has 3 major oxidation states or "valences": 0, the element; +2, ferrous, +3, ferric. 4. Possible compounds: Fe_2O_3 - hematite FeCl_3 FeO $\text{Fe}(\text{OH})_3$- insoluble complex ions $[\text{Fe}(\text{CN})_6]^{4-}$ $[\text{Fe}(\text{Phen})_3]^{2+}$

Module No:	Module Title: The Determination of Iron in Water
Approx. Time: 0.5 Hours	Submodule Title: Topic: Iron and Water
Instructional Objective: Upon completion of this module, the participant should be able to: 1. Describe the undesirable effects of high concentrations of iron in water supplies. 2. Name five ways in which iron is found in water. 3. Describe how ferrous iron can be converted to ferric iron in water samples. 4. Determine the oxidation state and solubility of iron under reducing and oxidizing conditions and various ranges of pH. 5. Differentiate between filtrable, colloidal, dissolved, and suspended iron. 6. List four precautions which should be taken when collecting samples for: (a) total iron, (b) ferrous iron, (c) filtrable iron determination. 7. Given the concentration of total iron, ferrous iron, and filtrable iron, calculate concentrations of suspended iron and ferric iron.	
Instructional Aids: Transparency Fe ²⁺ - List of ways iron can be found in water. Transparency Fe ³⁺ - pH and iron sample. Transparency Fe ⁴⁺ - Collection diagram	
Instructional Approach: Lecture - discussion	
References: Standard methods pp 208-210.	
Class Assignments: None	

Module No: Fe	Topic: Iron and Water
Instructor Notes:	Instructor Outline:
Transparency Fe2 Ways iron can be found in water	1. Undesirable effects: a. staining laundry and porcelain. b. bitter sweet astringent taste. c. contributes to hardness. d. rust in pipes. e. Maximum level 1 mg/l 2. Forms in water a. solution i. Fe^{2+} ii. Fe^{3+} b. colloidal - Fe(OH)_3 c. complexed d. organic 3. $\text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + e^-$ a. air b. other oxidants 4. pH and oxidizing conditions a. low pH (below 3) i. reducing Fe^{2+} soluble ii. oxidizing Fe^{3+} soluble b. high pH (above 3) i. reducing Fe^{2+} soluble ii. oxidizing Fe(OH)_3^- insoluble 5. Forms of Iron a. filtrable - dissolved; passes through filter b. colloidal - difficult to collect or filter c. dissolved - same as filtrable. d. suspended - non-filtrable collected on filter 6. Precautions a. total i. clean container ii. well mixed iii. don't let sample adhere to top iv. treat with acid b. filtrable as in a i-iii and filter immediately without exposure to air. c. ferrous as in a i-iv, prevent oxidation, sunlight. 7. Relationships: $\text{Fe}^{3+} = \text{Total} - \text{Fe}^{2+}$ $\text{suspended} = \text{total} - \text{filtrable}$
Transparency Fe3 pH and iron in water	
Transparency Fe4 Collection diagram	

Module No:	Module Title: The Determination of Iron in Water.
Approx. Time: 0.5 hours	Submodule Title: Topic: Spectrophotometry and the use of the Spectrophotometer.
Instructional Objective: Upon completion of this module, the participant should be able to: 1. Operate a spectrophotometer such as the Bausch and Lomb Spectronic 20; a. turn on and warm-up. b. set wave length. c. set zero d. set 100% T. 2. Determine the absorbance and transmittance of a colored sample - at a pre-determined wavelength. 3. Briefly explain how a spectrophotometer works. 4. Qualitatively, relate absorbance to deepness of color and concentration of solute. 5. Describe how wavelength and hue of color are related.	
Instructional Aids: Transparency Fe5- Condensed Spectrophotometer operating instructions. Transparency Fe6- Diagram of Spectrophotometer.	
Instructional Approach: Lecture / Demonstration; practice.	
References: Operator's manual for spectrophotometer used.	
Class Assignments: Read operators manual prior to session.	

Module No: Fe	Topic: Spectrophotometry
Instructor Notes:	Instructor Outline:
The instructor will need to have a spectrophotometer for demonstration and spectrophotometer(s) for student use. Transparency Fe5 Operating instructions Colored samples: dilute KMnO_4 or Fe-phenanthroline at 525 and 510 nm respectively. Transparency Fe6 Diagram of spectrophotometer	1. Review operating instructions. Have students prepare instrument for warm-up. 2. Determine absorbance of sample - Have students do same. How the spectrophotometer works. 3. Trace light path through spectrophotometer 4. Greater absorbance - deeper color, higher concentration. 5. Wavelength determines color of light absorbed, complement transmitted.

Module No:	Module Title: The Determination of Iron in Water
Approx. Time: 0.5 hours	Submodule Title: Topic: Beer's law and calibration curves

Instructional Objective:

Upon completion of this module, the participant should be able to:

1. Draw a Beer's law calibration curve given the appropriate experimental data.
2. Determine the slope of the curve and the value of k in the equation $C=kA$, where C is concentration and A is absorbance.
3. Given the value of A determine the value of C from a Beer's law curve or from the value of k in (2) above.

Instructional Aids:

Fe-7 - Graph paper transparency.

Instructional Approach:

Lecture/practice

References:

Standard Methods, pp. 10-13, 23-25.

Class Assignments:

None

Module No: Fe	Topic: Beer's Law and Calibration Curves												
Instructor Notes:	Instructor Outline:												
You will plot the curve on the transparency Fe7 with a ruler the students will follow. The following data is suggested. <table> <tr> <th>(x axis) conc. (mg/l)</th><th>(y axis) absorbance</th></tr> <tr> <td>10.0</td><td>0.11</td></tr> <tr> <td>20.</td><td>0.19</td></tr> <tr> <td>30.</td><td>0.30</td></tr> <tr> <td>40.</td><td>0.41</td></tr> <tr> <td>50.</td><td>0.49</td></tr> </table> This will have a slope of 0.01 $k = 100 \frac{1}{\text{mg}}$	(x axis) conc. (mg/l)	(y axis) absorbance	10.0	0.11	20.	0.19	30.	0.30	40.	0.41	50.	0.49	- curve - plot points - draw best straight line - slope $= \frac{\Delta y}{\Delta x}$ - or $\frac{y_1 - y_2}{x_1 - x_2} = \text{slope}$ $k = \frac{1}{\text{slope}}$ - determine value of C - directly from graph. - by proportions or substitution.
(x axis) conc. (mg/l)	(y axis) absorbance												
10.0	0.11												
20.	0.19												
30.	0.30												
40.	0.41												
50.	0.49												

Module No:	Module Title: The determination of Iron in Water
Approx. Time: 0.75 hours	Submodule Title: Topic: Principles of the iron/1, 10-phenanthroline spectrophotometric method.
Instructional Objective: Upon completion of this module, the participant should be able to: 1. Briefly describe how iron is determined spectrophotometrically 2. Describe how Iron (II) is distinguished from Iron (III) and how suspended iron is distinguished from filtrable iron. 3. Describe the color of the Fe^{2+}-1, 10 phenanthroline complex.	
Instructional Aids: Iron/Manganese video tape cassette.	
Instructional Approach: Television; lecture /discussion.	
References: Standard Methods, pp 208-213.	
Class Assignments: None	

Module No:	Topic: Principles of Determination
Instructor Notes:	Instructor Outline:
Fe/Mn Videotape Show tape up to the Mn part or entire tape if Mn analysis is included in this session.	1. Videotape viewing 2. Answers questions concerning tape. 3. Explain how the reducing step is omitted in Fe^{2+} analysis and how the analysis is performed on filtrate for filtrable Fe.

Module No:	Module Title: The Determination of Iron in Water
Approx. Time: 0.25 hours	Submodule Title: Topic: Lab Safety Review

Instructional Objective:

Upon completion of this module, the participant should be able to:

1. Locate the following in the laboratory and demonstrate proper use: emergency shower, fire extinguisher, eye wash, first aid kit.
2. Select and use safety glasses, lab coat or apron and gloves in the appropriate situation.
3. Describe the hazards associated with the chemicals used in the 1, 10-phenanthroline determination of iron.

Instructional Aids:

Hand-out of safety rules for the laboratory.

Instructional Approach:

Lecture/demonstration

References:

Basic laboratory skills module

Class Assignments:

Read lab safety rules.

Module No: Fe	Topic: Lab Safety Review
Instructor Notes:	Instructor Outline:
Hand out Lab Safety Rules to be determined locally	1. Show the location of and demonstrate use of various safety devices. 2. Point out the corrosive nature of concentrated laboratory acids and the effect on eyes.

Module No:	Module Title: The Determination of Iron in Water
	Submodule Title:
Approx. Time: 0.5 hours	Topic: Preparation of reagents and standards

Instructional Objective:

Upon completion of this module, the participant should be able to:

1. Prepare all solutions needed for the determination of, total iron and ferrous iron.
2. Prepare stock iron solution from ferrous ammonium sulfate or iron wire, standard iron solutions, and solutions for the calibration curve.
3. Calculate the concentration of each of the solutions in (2) above.
4. State which of the reagents will be used in each of the iron determinations.

Instructional Aids:

None

Instructional Approach:

Laboratory practice

References:

Standard Methods p.209-210.

Class Assignments:

Module No: Fe	Topic: Preparation of Reagents and Standards
Instructor Notes:	Instructor Outline:
a. $\text{NH}_2\text{OH} \cdot \text{HCl}$ 10g / 100 ml b. 250g $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ c. 0.1g $\text{C}_{12}\text{H}_8\text{N}_2 \cdot \text{H}_2\text{O}$ in 100 ml H_2O, 2 drops HCl d. 200g per 800 ml a. 20 ml con. H_2SO_4, 50 ml H_2O, 1.404g $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$; add KMnO_4 dilute to 1.1 b. i 50.0 ml stock/l ii 5 ml stock/l c. 2.0, 4.0, 6.0, 8.0, 10.0 ml per 100 ml flask	1. Have students prepare in groups: a. Hydroxylamine solution b. Ammonium acetate buffer c. phenanthroline solution d. Sodium acetate solution 2. Iron standards: a. Stock iron 200 μg per ml b. standard iron i 10 μg per ml c. calibration curve contains 20, 40, 60, 80, 100 μg Fe

Module No:	Module Title: The Determination of Iron in Water
Approx. Time:	Submodule Title:
0.5 hours	Topic: Preparation of standard curve

Instructional Objective:

Upon completion of this module, the participant should be able to:

1. Measure the absorbance of each calibration standard at the appropriate wavelength and record concentration, absorbance and wavelength for each sample.
2. Plot each point on the calibration curve and draw the appropriate straight line.

Instructional Aids:

None

Instructional Approach:

Laboratory practice

References:

Standard Methods, p.210

Class Assignments:

Module No: Fe	Topic: Preparation of Standard Curve
Instructor Notes:	Instructor Outline:
75 ml H ₂ O 10 ml phenanthroline solution, mix, let stand 10 min.	1. Treat each sample measured into flasks 2. Measure absorbance at 510 nm 3. Record μgFe, absorbance 4. Plot points 5. Draw graph

Module No:	Module Title: The Determination of Iron in Water
Approx. Time: 0.5 hours	Submodule Title:
	Topic: Testing of various samples

Instructional Objective:

Upon completion of this module, the participant should be able to:

1. Prepare samples for spectrophotometric measurement of total iron, filtrable iron and ferrous iron.
2. Measure and record the absorbance of each sample.
3. Determine the iron concentration in each sample from the absorbance and calibration curve.

Instructional Aids:

None

Instructional Approach:

Laboratory practice

References:

Standard Methods, pp. 210-211.

Class Assignments:

Module No:	Topic: Testing of Samples
Instructor Notes:	Instructor Outline:
Have student do a, b, or c For C: 50 ml sample, 2ml HCl, 1ml NH₂OH solution; boil to 20 ml, cool; add 10 ml buffer, 2 ml 1, 10 phenanthrodene, allow 15 min. for color. $\text{mg/l Fe} = \frac{\text{MoFe}}{100}$	1. Sample preparation: a. filtrable-filter into HCl b. ferrous-acidify c. total-mix well, reduce, heat d. add 1, 10 phenanthrodene 2. Read absorbance at 510 nm 3. Find absorbance on curve, determine μg iron present 4. Determine concentration of iron

Module No:	Module Title: The Determination of Iron in Water
Approx. Time: 0.5 hours	Submodule Title: Topic: Interferences
Instructional Objective: Upon completion of this module, the participant should be able to: 1. List various substances which may interfere with the determination of iron in water. 2. Describe measures which may be taken to minimize these interferences. 3. Describe in detail the determination of iron in the presence of organic interferences. 4. Estimate the accuracy and sensitivity of the method under various conditions.	
Instructional Aids: Transparency Fe8 - Chart of interferences. Transparency Fe9 - Diagram of determination in presence of organics.	
Instructional Approach: Lecture/discussion	
References: Standard Methods pp. 207-214.	
Class Assignments: None	

Module No: Fe	Topic: Interferences
Instructor Notes:	Instructor Outline:
Transparency Fe8 . Interferences	1. Interferences a. oxidizers b. CN^-, NO_2^-, PO_4^{3-} c. Cr, Zn, Co, Cu, Ni d. heavy metals e. color f. organics 2. Minimize interferences: a. Excess hydroxylamine b. Boiling with acid c. Excess phenanthroline d. Extraction e. Blank without 1, 10 phenanthroline 3. Organic Interference: a. Digestion b. Add to separatory funnel 10 ml, 15 ml conc HCl c. Extract with isopropyl ether (25 ml) d. Extract ether with water e. Reduce with NH_2OH, complex with 1, 10 phenanthroline 4. Precision, accuracy, sensitivity: a. 5% color comparison 3% spectrophotometric b. sensitivity 1 μgFe
Transparency Fe9 Determination in presence of Organics	

Module No:	Module Title: The Determination of Iron in Water
Approx. Time: 0.5 hours	Submodule Title: Topic: Test kit Method

Instructional Objective:

Upon Completion of this module the participant should be able to:

1. Determine the concentration of iron in a water sample using a commercially available test kit
2. Compare the results of the test kit method to those obtained by the phenanthroline method.

Instructional Aids:**Instructional Approach:**

Lecture/demonstration, laboratory practice.

References:

Instruction booklet for the test-kit used.

Class Assignments:

Module No: Fe	Topic: Test Kit Method
Instructor Notes:	Instructor Outline:
	1. Demonstrate the method of iron testing and comparison of colors to the students. 2. The students should perform the test on the same sample used in the phenanthroline method.

Exam Questions

The Determination of Iron in Water

A. Introduction

1. Write the atomic symbol for Iron
2. Iron is a(n)
 - a. non-metal
 - b. halogen
 - c. transition metal
 - d. noble gas
3. Which of the following is not a major oxidation state of iron
 - a. Fe
 - b. Fe⁶⁺
 - c. Fe³⁺
 - d. Fe²⁺
4. Give a proper formula for an iron compound.

Iron and water

5. True or False: "Rust" can be caused by iron in water.
6. True or False: Iron is sometimes found dissolved in water.
7. Name a substance which will convert Iron(II) to Iron(III).
8. Under what conditions of pH will Fe(OH)₃ dissolve?
9. Filtrable iron is the same as _____ iron.
10. In the analysis of what oxidation state of iron should care be taken to exclude air?
11. A sample contains 10 mg total iron, 5 mg ferrous iron. What is the mass of ferric iron present?

Spectrophotometry

12. For how many minutes should a B & L Spectronic -20 warm-up?
13. True or False: Absorbance is always the same as transmittance.
14. Briefly, what does a spectrophotometer measure?
15. The darker the color the _____ the absorbance.
16. Hue of color is related to:
 - a. wavelength
 - b. absorbance
 - c. transmittance.

Beer's Law and Calibration Curves

17. A _____ line between plotted points is said to be linear.
18. According to Beer's law, absorbance is _____ proportional to concentration.
19. A solution has an absorbance(A) of 0.2 and a proportionality constant, K, is 100 mg. If $C = KA$, calculate the concentration X
C of the solution.
20. What compound is used to make iron colored?
21. What compound is used to reduce Iron(III) to Iron(II)?
22. What color is the 1, 10 phenanthroline complex of iron?

Lab Safety Review

23. If acid is splashed in your eyes, what device should be used to rinse it out?
24. Should you wear safety goggles when you are handling concentrated hydrochloric acid?
25. Name a hazardous substance used in the analysis of iron.

Preparation of Reagents and Standards

26. What two substances are used in the preparation of an acetate buffer solution?
27. Iron wire or _____ can be used to prepare a standard iron solution.
28. 2.0 ml of a stock solution in which 1 ml=10 μ g Fe is diluted to the mark in a 100 ml volumetric flask. Calculate the concentration of the resulting solution in mg/l.
29. Hydroxylamine is not used for the determination of _____ iron.

Preparation of Standard Curve

30. Each Data point on a calibration curve consists of two pieces of data. What are they?
31. The line of a calibration curve.
 - a. connects all the points
 - b. is the best straight line which passes between the points
 - c. is a curved line through all the points

Testing of Various Samples

32. Prior to the spectrophotometric measurement how must a sample be handled for the determination of filtrable iron?
33. What instrument is used to determine the absorbance of a sample.
34. A 50 ml sample of water contains 100 μ g of iron. Calculate the iron concentration in mg/liter.

Interferences

35. Which of the following substances will interfere with the determination of iron in water?
 - a. oxidizing agents
 - b. cobalt in excess of 5 mg/l
 - c. nitrite
 - d. cyanide
 - e. all the above
36. Adding excess hydroxylamine will eliminate errors caused by what type of interference?
37. What must be done to a sample to eliminate interferences due to organic substances?
38. The minimum detectable quantity of iron using the spectrophotometer with a 1-cm cell is:
 - a. 0.1 g
 - b. 2 picograms
 - c. 1 mg
 - d. 50 micrograms

THE DETERMINATION OF IRON IN WATER

EQUIPMENT AND SUPPLIES LIST

EQUIPMENT

- | | |
|----------------------------------|--|
| 1. spectrophotometer | 12. 400 ml beaker |
| 2. 2 absorbance cells | 13. liter volumetric flask |
| 3. safety goggles | 14. 1, 2, 5, 10, 50 ml volumetric pipets |
| 4. apron | 15. 125 ml erlenmeyer flask |
| 5. 10 - 100 ml volumetric flasks | 16. 45 mm membrane filter with holder |
| 6. laboratory balance | 17. vacuum flask |
| 7. 1000 ml beaker | 18. 2 - 1000 ml glass bottles |
| 8. ring stand | 19. 100 ml graduated cylinder |
| 9. iron ring | 20. boiling beads |
| 10. wire gauze | 21. 2000 ml beaker |
| 11. bunsen burner | 22. graph paper |

CHEMICALS

1. concentrated hydrochloric acid
2. hydroxylamine hydrochloride
3. sodium acetate trihydrate
4. 1, 10 phenanthroline
5. standard grade ferrous ammonium sulfate hexahydrate
6. 0.1 N $KMnO_4$
7. distilled water
8. ammonium acetate
9. glacial acetic acid
10. concentrated sulfuric acid

The Determination of Iron in Water

Laboratory Procedure

I. Preparation of Standards and Reagents

- A. Obtain all the equipment, supplies, and chemicals listed on "equipment" hand-out. Turn on spectrophotometer for warm-up.
- B. Prepare the following solutions:
 1. Hydroxylamine: Add 10.0 g hydroxylaminehydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) to a 100 ml volumetric flask; dilute to the mark with distilled water. Label the flask. Mix by repeated inversion of stoppered flask.
 2. Ammonium Acetate Buffer: Add 250g ammonium acetate ($\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$) to a 2000 ml beaker. Add 150 ml water to the beaker; mix to dissolve. To this solution add 700 ml glacial acetic acid. Stir to make a uniform solution. Transfer to a 1000 ml glass stoppered bottle. Label the bottle.
 3. Sodium Acetate solution: Add 200 g sodium acetate trihydrate ($\text{NaC}_2\text{H}_3\text{O}_2\cdot 3\text{H}_2\text{O}$) to a clean 2000 ml beaker. Add 800 ml distilled water to the beaker. Mix to dissolve. Transfer to a 1000 ml glass bottle and label.
 4. Phenanthroline Solution: Add 0.1 g 1.10 phenanthroline monohydrate ($\text{C}_{12}\text{H}_8\text{N}_2\cdot\text{H}_2\text{O}$) to a 150 ml beaker. Add 100 ml distilled water. Stir to mix. Add 2 drops concentrated hydrochloric acid (HCl). Stir to dissolve. Transfer to a 150 ml glass-stoppered bottle; label the bottle.
- C. Prepare the following stock standard iron solutions.
 1. Stock iron (200 micrograms per ml) with stirring. (Make sure you are wearing safety glasses), add 20 ml concentrated sulfuric acid (H_2SO_4) to 50 ml distilled water in a 150 ml beaker. After the solution has cooled, add exactly 1.404g standard grade ferrous ammonium sulfate hexahydrate ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2\cdot 6\text{H}_2\text{O}$) which has been weighed on an analytical balance. Stir to dissolve. Add several drops of 0.1 N KMnO_4 solution to the iron solution until a faint pink color remains. Add the iron solution to a 1 l volumetric flask. Rinse the contents of the breaker into the flask with distilled water. Fill the flask to the mark with distilled water. Mix by repeated inversion. Transfer to a liter bottle and label the bottle.

2. Standard iron (10 micrograms per ml)

With a volumetric pipet, add 50 ml of the solution in 1. to a 1 liter volumetric flask and dilute to the mark with distilled water.

II. Preparation of Beer's law calibration curve

A. Label five 100 ml volumetric flasks as follows:

20 μ g Fe, 40 μ g Fe, 60 μ g Fe, 80 μ g Fe, 100 μ g Fe.

Add respectively with volumetric pipets: 2.0, 4.0, 6.0, 8.0, 10.0 ml standard iron solution to each flask. To each flask add 1 ml hydroxylamine solution and 1 ml sodium acetate solution. Fill each flask about 3/4 full with distilled water. Add 10 ml phenanthroline solution to each flask. Dilute to the mark with distilled water, mix, and allow to stand for 10 minutes.

B. Set the spectrophotometer wavelength to 508nm. With the sample compartment empty set 0T. Insert reagent blank, set 100% T. Transfer portions of each solution above into 5cm absorption cells. Measure the absorbance of each solution and record on data sheet.

C. Prepare calibration curve by labeling the x-axis " μ gFe", the y-axis "absorbance" and inserting values 0-1.0 on the y axis, 0-100 on the x-axis. Plot the data points. With a straightedge, draw the best straight line through the origin and the points. Title the graph "Beer's Law Calibration Curve for Iron."

III. Preparation of Sample

Choose one of the following methods

A. Total iron

Collect a representative sample. Add 2ml-HCl to the sample bottle to dissolve suspended iron. At the time of analysis mix the sample and add precisely 50ml to a 125ml eulynmeyer flask. Add 2ml concentrated HCl and 1 ml hydroxylamine solution. Add boiling beads and heat with bunsen burner to boiling. Boil until about 1/3 of the solution remains. Remove from heat, allow to cool to room temperature and transfer to a 100 ml volumetric flask. Add 10ml buffer solution and 2ml phenanthroline solution. Dilute to the mark with distilled water. Mix.

B. Filtrable iron.

At the time of collection or soon thereafter, pour a well mixed 100 ml sample through a 0.45-mm membrane filter into a vacuum flask which contains 1 ml concentrated HCl/. Transfer 50 ml of this sample

to a 125 ml erlynmeyer and treat as in "1" above.

C. Ferrous iron

Collect a representative sample. Add 2ml HCl for each 100 ml sample. Fill the bottle completely and stopper immediately. Immediately before analysis, pipet 50ml into a 100 ml volumetric flask. Add 20 ml phenanthroline solution and 10 ml buffer solution. Mix well. Dilute to the mark with distilled water.

IV. Measurement and calculation

- A. Measurement for total or filterable iron; wait 15 minutes prior to measurement. For ferrous iron measure within 10 minutes. Transfer sample to 5 cm absorption cell. Set wavelength at 508 nm. Set OT with sample compartment empty. Set 100% T with reagent blank. Insert sample; read absorbance; record absorbance.
- B. Interpolation
From the calibration curve and the sample absorbance, determine the micrograms iron present in the sample.
- C. Divide the number in B by 50.0 to determine the mg/l concentration.
- D. Comment on possible sources of error or other unusual problems in the analysis including sample collection, storage, pretreatment.

Data Sheet for Iron Analysis

Sample number _____

Calibration curve

ml standard	$\mu\text{gFe}/100 \text{ ml}$	absorbance
2.0	20	_____
4.0	40	_____
6.0	60	_____
8.0	80	_____
10.0	100	_____

Attach graph paper with plotted curve.

Sample to be analyzed for (check one):

_____ total iron
_____ filtrable iron
_____ ferrons iron

Absorbance of sample _____

Micrograms Fe present _____

Fe concentration $\text{mg/l} = \frac{\mu\text{gFe}}{50}$
= _____

Comments:

Analyst _____

Date _____

PERIODIC TABLE OF THE ELEMENTS

Atomic weight values listed in parentheses are approximate.

TRANSPARENCY FE2

Ways in Which Iron is Found in Water

I. Solution.

A. Fe^{2+} , iron(II), ferrous.

B. Fe^{3+} , iron(III), ferric.

II. Colloidal- Iron(III) hydroxide, $\text{Fe}(\text{OH})_3$

III. Complexed

IV. Organic

TRANSPARENCY FE3

Forms of Iron
Under Various Conditions of pH

	Low pH (less than 3)	High pH (Greater than 3)
Reducing conditions	Fe^{2+} soluble	Fe^{2+} soluble
Oxidizing conditions	Fe^{3+} soluble	$\text{Fe}(\text{OH})_3$ insoluble

TRANSPARENCY FE4

Sample Collection

For all iron

wash collection bottle
with acid and distilled
water

agitate well

filtrable

filter sample
immediately

analyze

ferrous

acidify at
time of
collection

avoid sunlight

analyze

total

well mixed
agitated
sample

treat with
acid

analyze

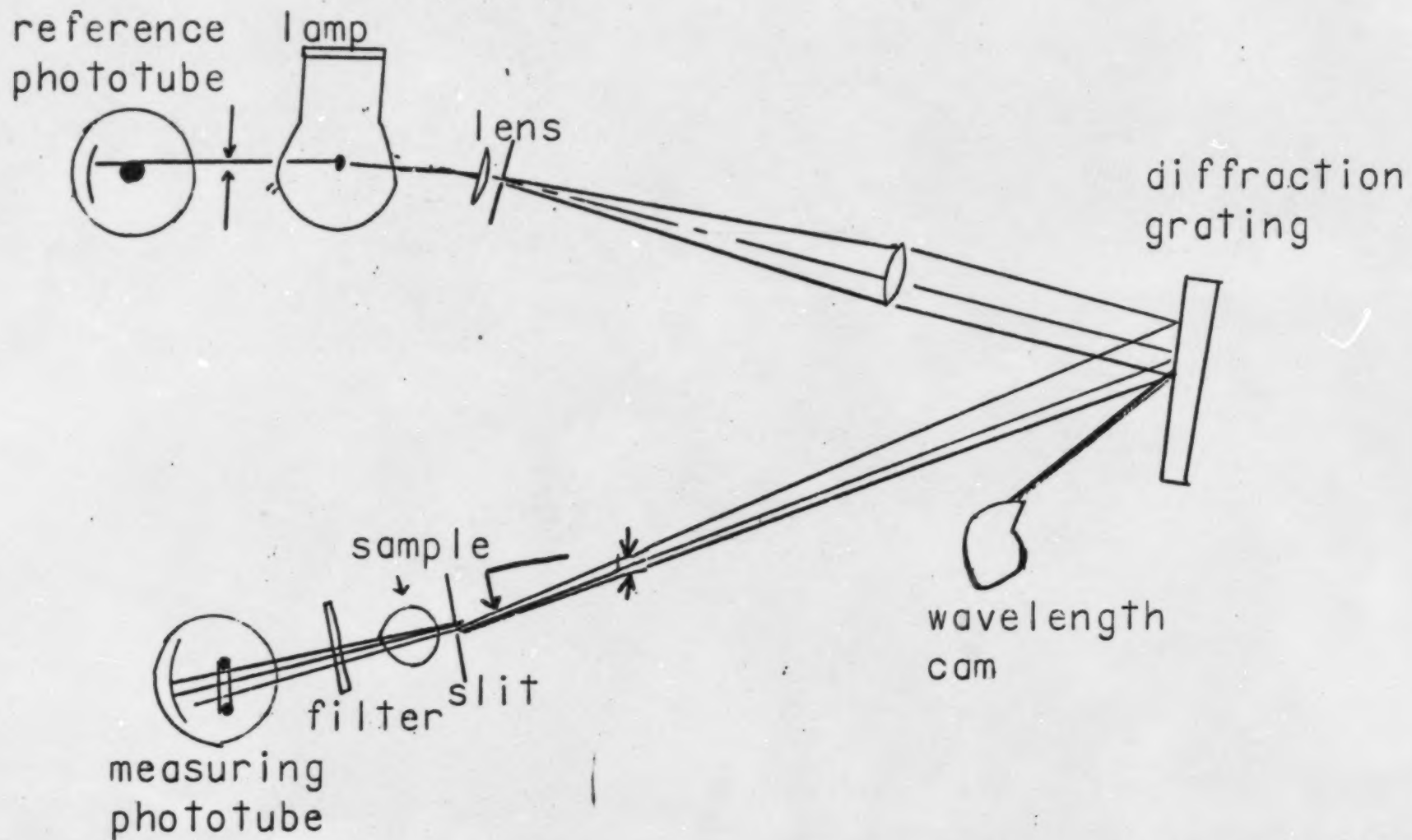
TRANSPARENCY FE5

Condensed Spectrophotometer Operating instructions

1. Turn on- warmup 15 min.
knob left front
2. Set zero
knob left front
3. Set wavelength
knob top right
4. Insert blank
sample compartment
5. Set 100% T - full scale
knob right front
6. Insert unknown
sample compartment
7. Read absorbance or %T

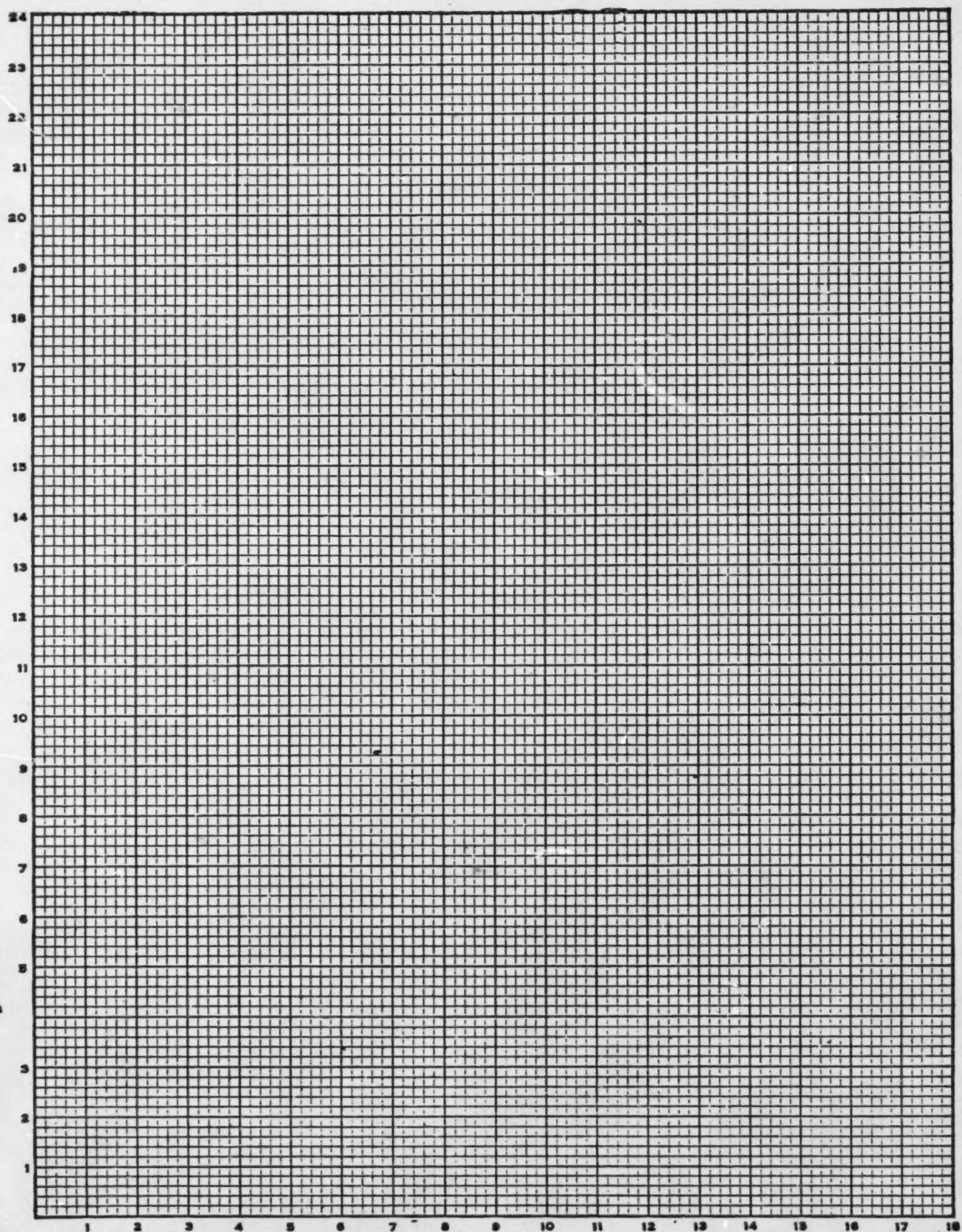
TRANSPARENCY FE6

Spectrophotometer diagram



Transparency FE7

Graph paper for Calibration Curve



TRANSPARENCY FE8

Chart of Interferences

Interference	To reduce interference
1. oxidizers	excess hydroxylamine
2. CN^- , PO_4^{3-} , NO_3^-	boiling
3. Metal ions; Cr, Zn, Co, Cu, Ni.	excess phenanthroline
4. Color	blank of sample processed without phenanthroline-
5. Organics	Digestion-extraction

TRANSPARENCY FE9

